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Indian Standard

SPECIFICATION FOR MANGANESE CARBONATE

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Indian Standard
SPECIFICATION FOR MANGANESE CARBONATE

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Indian Standard

SPECIFICATION FOR MANGANESE CARBONATE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 28 March 1985, after the draft finalized by the Inorganic Chemicals (Misc) Sectional Committee had been approved by the Chemical Division Council.

0.2 Manganese carbonate is used in the manufacture of manganese salts, medicine, paint pigment, fertilizers and in food additives.

0.3 It does not cover manganese carbonate used as food additives, fertilizer and in pharmaceutical preparations.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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1. SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for manganese carbonate.

2. REQUIREMENTS

2.1. Description — The material shall be in the form of light brown to brown homogeneous powder. It shall be free from foreign matter.

2.2 The material when tested as prescribed in Appendix A shall also comply with the requirements given in Table 1. Reference to relevant clauses of Appendix A is given in col 4 of Table 1.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in polyethylene laminated jute bags or as agreed to between the purchaser and the supplier.

*Rules for rounding off numerical values (*revised*).

TABLE 1 REQUIREMENTS FOR MANGANESE CARBONATE

(*Clause* 2.2)

SL NO.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST (REF TO CL. NO. IN APPENDIX A)
(1)	(2)	(3)	(4)
i)	Assay (as $MnCO_3$), percent by mass, *Min*	92·0	A-1
ii)	Nitric acid in solubles, precent by mass, *Max*	0·15	A-2
iii)	Alkali carbonates (as Na_2CO_3), percent by mass, *Max*	0·01	A-3
iv)	Chlorides (as Cl), percent by mass, *Max*	0·1	A-4
v)	Sulphates (as SO_4), percent by mass, *Max*	2·5	A-5
vi)	Calcium (as Ca), percent by mass, *Max*	0·2	A-6
vii)	Iron (as Fe), percent by mass, *Max*	0·1	A-7

3.2 Marking — Each package shall bear legibly and indelibly the following information :

a) Name of the material;

b) Name of the manufacturer and his recognized trade-mark, if any;

c) Gross and net mass;

d) Date of manufacture; and

e) Batch number.

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the reqnirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 The method of drawing representative samples of the material and the criteria for conformity shall be as prescribed in Appendix B.

APPENDIX A

(*Clause* 2.2)

METHOD OF TEST FOR MANGANESE CARBONATE

A-0. QUALITY OF REAGENTS

A-0.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-1. ASSAY

A-1 1 Reagents

A-1.1.1 *Concentrated Nitric Acid — see* IS : 264-1976†.

A-1.1.2 *Sodium Bismuthate* — powder.

A-1.1.3 *Ferrous Ammonium Sulphate Solution* — 0·1 N (approximately).

A-1.1.4 *Standard Potassium Permanganate Solution* — 0·1 N.

A-1.2 Procedure — Weigh accurately about 1 g of the sample into a 250-ml volumetric flask containing about 50 ml of water and dissolve it therein by slowly adding 5 ml of nitric acid. When solution is complete make up to 250 ml with water. Transfer 25 ml of it to 500 ml iodine flask containing an ice cooled mixture of 75 ml of water and 20 ml of nitric acid, add 1·5 g of sodium bismuthate and shake gently for 2-3 minutes. Dilute with 100 ml of water washing down the sides and filter through a sintered glass funnel. Wash the flask and funnel with minimum quantity of nitric acid diluted with 33 volumes of water until the washing is colourless. To the filtrate and washings collected in a 500-ml conical flask, add 50ml of ferrous ammonium sulphate and titrate the excess of it with standard potassium permanganate. Find out the blank reading by titrating 50 ml of the ferrous ammonium

*Specification for water for general laboratory use (*second revision*)

†Specification for nitric acid (*second revision*).

sulphate mixed with a cooled mixture of 100 ml of water and 20 ml of nitric acid with standard potassium permanganate solution.

A-1.3 Calculation

$$\text{Assay (as } MnCO_3\text{), percent by mass} = \frac{(V_1 - V_2) \times N \times 23}{M}$$

where

V_1 = volume in ml of standard potassium permanganate solution required for the blank;

V_2 = volume in ml of standard potassium permanganate solution required for sample;

N = normality of standard potassium permanganate solution, and

M = mass in g of the material taken for the test.

A-2 NITRIC ACID INSOLUBLES

A-2.1 Reagents

A-2.1.1 *Dilute Nitric Acid* — 20 percent (*m/v*).

A-2.2 Procedure — Dissolve 5·0 g of the sample in 100 ml of dilute nitric acid, filter, wash the insoluble residue with hot water till filtrate is free from acid and ignite in a tared crucible and weigh to constant mass.

A-2.3 Calculation

$$\text{Nitric acid insolubles, percent by mass} = M \times 20$$

where

M = mass in g of the residue+crucible.

A-3 ALKALI CARBONATE

A-3.1 Reagents

A-3.1.1 *Standard Hydrochloric Acid* — 0·02 N.

A-3.2 Procedure — Boil 2 g of the sample with 30 ml of water, filter, wash with 20 ml of hot water and cool. Any pink colour produced in the filtrate when 2 drops of phenolphthalein are added should not require more than 0·2 ml of standard hydrochloric acid to discharge it.

A-4. CHLORIDES

A-4.1 Reagents

A-4.1.1 *Dilute Nitric Acid* — 30 percent (*v/v*).

A-4.1.2 *Silver Nitrate Solution* — 10 percent (*m/v*).

A-4.1.3 *Standard Chloride Solution* — Dissolve 0·164 9 g of ignited sodium chloride in 1 000 ml of water. Dilute 10 ml of the solution to 100 ml. One millilitre of the diluted solution is equivalent to 0·01 mg of chloride (as Cl).

A-4.2 Apparatus

A-4.2.1 *Nessler Cylinder* — 50-ml capacity.

A-4.3 Procedure — Dissolve 1 g of the sample in 20 ml of dilute nitric acid, filter and make up to 100 ml. To 5 ml of it, taken in a Nessler cylinder, add 3 drops of silver nitrate solution, dilute and mix well. Any turbidity produced should be not greater than that produced in a control where 5 ml of standard chloride solution containing 0·01 mg of chloride per ml were acidified with 1 ml of dilute nitric acid and treated in a similar manner.

A-5. SULPHATES

A-5.1 Reagents

A-5.1.1 *Hydrogen Peroxide Solution* — 30 percent.

A-5.1.2 *Hydrochloric Acid — see* IS : 265 - 1976*.

A-5.1.3 *Dilute Hydrochloric Acid* — 10 percent (*v/v*).

A-5.1.4 *Barim Chloride Solution* — 10 percent (*m/v*).

A-5.1.5 *Standard Sulphate Solution* — Dissolve 1·48 g of ignited sodium sulphate (Na_2SO_4) in water and dilute to 1 000 ml. Take 10 ml of this solution and dilute to 100 ml. One millitre of this solution contains 0·1 mg of sulphate (as SO_4).

A-5.2 Procedure — To 5 g of the sample in a 250-ml beaker, add 25 ml of water, 2 ml of hydrogen peroxide and heat on a steam bath for 15 minutes. Add 15 ml of hydrochloric acid, evaporate to dryness on a steam bath, dissolve the residue in 40 ml of water and 1 ml of hydrochloric acid, filter if necessary and dilute to 100 ml in a volumetric flask Dilute 10 ml of this to 100 ml and preserve the rest.

*Specification for hydrochloric acid (*second revision*).

A-5.3 To 2 ml of the above diluted solution transferred into a 50 ml Nessler cylinder add 1 ml of 10 percent hydrochloric acid followed by 2 ml of barium chloride solution, stir well. Any turbidity produced should not be greater than that produced in a control where 0·25 mg of sulphate is treated in a similar manner.

A-6 CALCIUM

A-6.1 Reagents

A-6.1.1 *Ammonium Chloride Solution* — 11 percent (*m/v*).

A-6.1.2 *Dilute Ammonia Solution* — 10 percent (*v/v*).

A-6.1.3 *Standard Calcium Solution* — Weigh 0.25 g of calcium carbonate dried at 120°C and dissolve it in the minimum quantity of dilute hydrochloric acid. One millilitre of the solution is equivalent to 0·1 mg of calcium (as Ca).

A-6.1.4 *Ammonium Oxalate Solution* — 4 percent (*m/v*).

A-6.2 Procedure — To 2 ml solution from **A-5.1**, transferred into a 100 ml beaker and diluted with 10 ml of water, add 2 ml of ammonium chloride (11 percent) followed by 10 ml (or more if necessary) of dilute ammonia and 7 ml of dilute hydrogen peroxide (1:6) with stirring. Boil to complete the precipitation, filter and wash the precipitate into a 50-ml Nessler cylinder where it is cooled to room temperature. Add 4 ml of ammonium oxalate solution (4 percent dilute to 50 ml, stir with a glass rod and let it stand for 20 minutes. Any turbidity produced should be not greater than that produced in a control where 0·2 mg of freshly prepared calcium were treated with 2 ml of ammonium chloride, 2 ml of dilute ammonia and 4 ml of ammonium oxalate in 50 ml volumes 20 minutes earlier.

A-7. IRON

A-7.1 Reagents

A-7.1.1 *Concentrated Hydrochloric Acid — see* IS : 265 - 1976*.

A-7.1.2 *Butanolic Potassium Thiocyanate Solution* — Dissolve 10 g of potassium thiocyanate in 10 ml of water. Add sufficient *n* butanol to make up to 100 ml and shake vigorously until the solution is clear.

A-7.1.3 *Standa d Iron Solution* — Dissolve 0·702 g of ferrous ammonium sulphate [$FeSO_4 (NH_4)_2 S0_4 6H_2 O$] in water and add 10 ml of concentrated hydrochloric acid and dilute with water to 1000 ml. One millilitre of the solution is equivalent to 0·1 mg of iron (as Fe).

*Specification for hydrochloric acid (*second revision*)

A-7.2 Procedure — To 0·5 ml from **A-5.1** into a test tube add 10 ml of water, 1 ml of hydrochloric acid followed by 15 ml of butanolic potassium thiocyanate solution. Shake well. The red colour produced in the butanolic layer should not be more than 5 ml of standard iron solution prepared as under when treated as per the aliquot.

A-7.2.1 Pipette 5 ml of standard iron solution containing 0·5 mg of Fe into a beaker, add 20 ml of water, 2 ml of 30 percent hydrogen peroxide and heat in a steam bath for 15 minutes. Add 15 ml of hydrochloric acid and evaporate to dryness. Dissolve the residue in 40 ml of water and 1 ml of HCl. Filter if necessary and dilute to 100 ml. Take 5 ml of this standard solution and treat as the aliquot.

APPENDIX B

(*Clause* 4.1)

SAMPLING OF MANGANESE CARBONATE

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Samples shall not be taken at the place exposed to adverse affects of weather.

B-1.2 The sampling instruments and sample containers shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.5 The samples shall be placed in clean, dry and airtight glass or other suitable containers on which the material has no action.

B-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.7 Each sample container shall be sealed airtight after filling and marked with full details of sampling the date of sampling and the lot and batch numbers.

B-2. SCALE OF SAMPLING

B-2.1 Lot — All the containers in a single consignment of the material drawn from the same batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture the containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

B-2.2 Samples shall be tested for each lot for ascertaining the conformity of the material to the requirements of this specification.

B-2.3 The number of containers (n) to be chosen from a lot shall depend upon the size of the lot (N) and shall be in accordance with Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

Lot Size	Sample Size
Up to 50	3
51 ,, 100	4
101 ,, 150	5
151 ,, 300	7
301 and above	10

B-2.4 These containers shall be chosen at random from the lot. In order to ensure the randomness of selection, reference may be made to IS : 4905-1968*.

B-3. NUMBER OF TESTS

B-3.1 Test for the determination of assay shall be conducted on individual sample.

B-3.2 Test for the determination of all other characteristics given in Table 1 shall be conducted on composite sample.

B-4. CRITERIA FOR CONFORMITY

B-4.1 For Individual Sample — From the test results, the average and the range shall be calculated as follows:

$$\text{Average} = \frac{\text{Sum of the test results}}{\text{Number of tests}}$$

*Methods for random sampling.

Range = The difference between the maximum and the minimum values of the test results.

The lot shall be declared as conforming to the requirements of assay if $\bar{X} - 0{\cdot}6\ R$ is greater than the minimum value specified.

B-4.2 Composite Sample — For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample, the test result for each characteristic shall satisfy the relevant requirements given in Table 1.

(*Continued from page* 2)

Members	*Representing*
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